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(54) Title: CARBONLESS COPY PAPER

(57) Abstract

A coating composition for the preparation of CB coated carbonless copy paper includes a water based dispersion of a sizing agent which is such as to increase the hydrophobicity of the paper substrate. Sizing agents that are suitable include, alkyl ketene dimers and rosin derivatives, and the use of such substances has been found to reduce the measured through-thickness porosity of substrate papers with respect to the solvents employed for the colour-formers, thereby making it possible to utilise grades of paper previously considered unsuitable for use in the manufacture of carbonless copy materials. Alkyl ketene dimers that may be used include those with C₁₂-C₂₂ alkyl radicals, and the rosins which may be used include fortified rosins and modified rosin derivatives.

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Title: Carbonless Copy Paper**Description of Invention**

This invention relates to so-called carbonless copy paper, in which a sheet of paper substrate is provided on its reverse face with a coating, known as a CB coating, containing droplets of a solution of colour-formers which are themselves normally substantially colourless, and in use the CB coating is brought into contact with a receptor sheet of paper substrate which is provided on its front face with a coating, known as a CF coating, of a material which reacts with such colour-formers to produce a colour when the colour-former solution is released from the CB coating and onto the CF coating of the second sheet. Such carbonless copy paper is made in CB (coated back) and CFB (coated front and back) grades, and the present invention applies to both such grades.

For the manufacture of such carbonless copy paper many technical parameters within the specification for the paper substrate need to be tightly controlled in order to provide the archival requirements of long term stability and image permanence. One of the main concerns regarding the control of the paper substrate specification revolves around the control of its porosity insofar as this affects the transfer of colour-former solution, after its release from the coating, through the paper from one face to the other.

During the manufacture, printing, finishing and subsequent handling of carbonless paper having such a CB coating, that is CB and CFB grades, a small proportion of the droplets of the colour-former are prematurely released, for example by inadvertent rupture of individual capsules in which the solution is confined in the CB coating, thus giving rise to the presence of capsule core

material (i.e. free solvent comprising oil, and colour-former, comprising reactive dyestuff) in the paper substrate.

Although most manufacturers of carbonless copy papers make additions to the CB coatings either to protect against premature capsule breakage (e.g. addition of calibrated wheat starch) or to provide for the absorption of any resultant free oil (e.g. addition of cellulose fibres) nevertheless it appears that a proportion of the released capsule core material is absorbed into the paper substrate and, in time, can permeate through the thickness of the substrate.

This is in the context of paper - general, not CS particularly. Does not obviously relate to coated papers. The overall teaching is that high porosity is bad

The more commonly used measurements of paper substrate porosity (e.g. Bendtsen Porosity/Gurley) use, in one form or another, measurements of the resistance to the flow of air through the paper as a measure of air permeability to decide whether the base paper has low porosity (i.e. less than 200 mls/min Bendtsen) or high porosity (i.e. greater than 1000 mls/min). A further test, the Water Cobb test, is also commonly used to determine the surface absorbency of the base sheet to aqueous coatings. In the manufacture of carbonless substrate papers a Water Cobb value below 20 gsm/60 secs is commonly specified.

Whilst the air permeability type porosity values and Water Cobb surface absorbency figures provide useful guidelines as to the suitability of paper substrates for conversion into carbonless coated grades, it has become apparent that specification of these parameters is not entirely sufficient to protect the resultant product against long term instability and to ensure long term image legibility.

We have concluded that some paper substrates which meet the normally acceptable standards of low air permeability type porosity and Water Cobb values can nevertheless exhibit unacceptably high permeation by capsule core material, and the term "through thickness porosity" is used herein to describe the resistance of the paper substrate to the through-thickness migration of

capsule core materials such as are released by intentional or premature capsule breakage

Investigations have shown that the disadvantages arising from the use of paper substrates which exhibit poor "through thickness porosity" include background discolouration of CFB grades and reduced image legibility.

The first of these disadvantages, background discolouration of CFB grades, is due to the through-thickness migration of the prematurely released capsule core material (free oil and dyestuffs) resulting in a colour reaction with the co-reactant CF coating applied to the opposite (front) side of the same sheet.

The use of thin layer chromatography is a well known technique used for the separation and identification of individual colour-formers contained within the blends of colour-formers conventionally used for the development of "black image" carbonless copy paper. This identification method is based on the fact that individual colour-formers have different rates of migration and therefore, in a fixed time period, become separated, thereby enabling identification. The rate and distance travelled by each dyestuff is referred to within the analysis technique as the "R.F. value" and colour formers with a high R.F. value separate faster and further than those with a low R.F. value.

When using a paper substrate of poor "through thickness porosity", the background discolouration of the co-reactant can vary with time (e.g. over a period of 3 to 6 months) from, for example 'pink' through 'purple' to 'black'. It is therefore reasonable to assume that chromatographic separation of the individual colour-formers by the filler and fibres of the paper substrate of poor "through thickness porosity" is the most likely mechanism for the development of background discolouration. This discolouration is not immediate but develops over a period of several months, both before and after use, consequently a product which is apparently satisfactory at the time of manufacture can be totally unacceptable after storage for several months, either before or after use.

The second disadvantage, reduced image legibility, results from excessive image spread on CFB grades which adversely affects image permanence. The development of the image in a multipart set of carbonless copy paper is based on the breakage of capsules on the reverse of the sheet by pressure applied to the upper surface. Although most of the released capsule core material is absorbed by, and reacts with, the adjacent co-reactant CF coating to develop the image, it has been established that a proportion of the released core material remains within the CB coating. When using a substrate of poor "through thickness porosity" the non-absorbed fraction of the released core material will migrate through the paper substrate by a mechanism similar to capillary action, and this results, with time, in the phenomenon, sometimes called "strike-back", of broadening of the image on the upper CF coated surface of the CFB sheet, thus adversely affecting the image legibility on storage.

A further related phenomenon observed is that, as a direct result of the excess oil and colour-formers present when such "strike-back" phenomenon occurs, a partial chromatographic separation of the individual colour-formers can occur on the upper surface of the CFB sheet resulting in a "halo" effect around the image on the upper surface, referred to as "ghosting" of the image, which further degrades image legibility.

It is accordingly the object of the invention to reduce such effects by the inclusion of suitable additives in the capsule coating so that paper substrates of inherently poor "through thickness porosity" can be utilised with improved long term stability and image legibility.

According to the invention we provide a coating composition for the preparation of CB coated carbonless copy paper characterised by the inclusion an additive to increase the resistance to penetration of the substrate paper by the non-aqueous solvents as used for the colour-formers employed in the CB coating.

Surprisingly, we have found that this function can be provided by means of an additive which comprises a water based dispersion of a sizing agent which is such as to increase the hydrophobicity of the paper substrate.

Typical solvents employed in the preparation of solutions of colour-formers include oils such as chloroparafins, di-isopropylnaphthalene, animal and vegetable oils, mineral oils, and yet the addition of hyrdophobicity increasing materials to the CB coating composition has been found to reduce penetration of the paper by such oils.

In particular, sizing agents that are suitable include, alkyl ketene dimers and rosin derivatives.

The use of such substances has been found to improve, that is to say reduce, the measured through-thickness porosity of substrate papers, making them more suitable for use in the manufacture of carbonless copy materials.

Alkyl ketene dimers that may be used in accordance with the invention include those in which the alkyl radicals thereof generally contain 12 to 22 carbon atoms, typically 14 to 18, and in particular mixtures thereof as are commercially available. While the alkyl radicals are typically straight-chain, i.e. primary alkyl, and saturated, it is possible to use dimers which are at least partly composed of secondary alkyl radicals, or at least partly composed of unsaturated alkyl radicals.

The dimer may be stabilised in emulsion form preferably by dispersion in a cationic starch, for example derived from potato, maize, wheat, etc. The ratio of dry dimer to dry starch may be generally between 10:1 to 1:2.

A further additive which may be included in such AKD/starch emulsions is a cationic promoter, such as dicyandiamide formaldehyde, polyamides, polyamines, etc. which act as catalysts to increase the rate of reaction between the dimer and the anionic paper fibres of the substrate. The dry ratio of such promoters to dimer may generally be in the ranges 1:10 to 1:1.

Rosin is a complex molecule developed from wood and a by product of the pulp industry. Rosin is composed of blends of many organic acids principally different derivatives of abietic acid (e.g. abietic acid itself; neoabietic acid; dihydro and tetra hydro abietic acid) and pimaric acid (e.g. pimaric acid itself, levo pimaric acid, iso-pimaric etc), and the rosins which may be used in accordance with the invention particularly include fortified rosins and modified rosin derivatives. Fortified rosins may be produced by the reaction of rosin acid with either fumaric acid or maleic anhydride. The modified rosin derivatives that may be used include rosin esters, of which penta erythritol hydrogenated rosin ester is a particular example.

Dispersions of such rosins are preferably stabilised in cationic starches or wet strength resins, such as polyaminoamides, typically in a rosin to dry stabiliser ratio of 4:1 to 12:1.

We have found that a result of the inclusion of such additives to the capsule coating formulation, paper substrates of poor "through thickness porosity" can be used to produce grades of acceptable archival properties, thus extending the source of suitable substrate papers to more readily available, and less expensive, grades of papers, such as continuous stationery grades (e. g. constats) and photocopier grade papers.

The invention further resides in a carbonless copy paper having a CB coating thereon characterised by the presence of an additive to increase the resistance to penetration of the substrate paper by the non-aqueous solvents as used for the colour-formers employed in the CB coating, more particularly a water based dispersion containing a sizing agent which is such as to increase the hydrophobicity of a paper substrate treated therewith, preferably either an alkyl ketene dimer, with or without the addition of promoters as outlined above, or a modified rosin, particularly fortified rosins and rosin derivatives chemically modified and stabilised as outlined above.

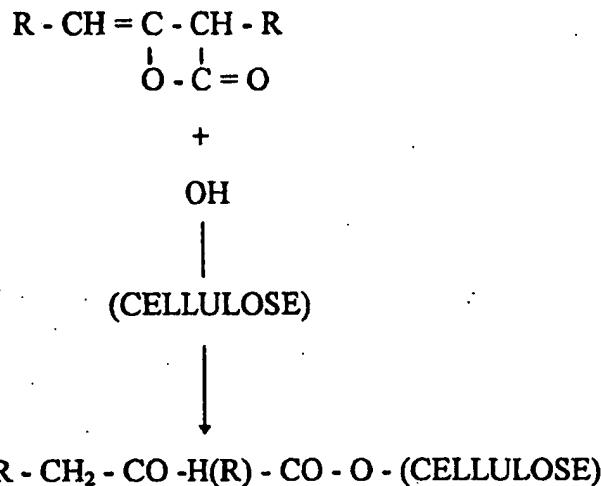
The additive is preferably incorporated in the CB coating composition which is applied to the paper, but alternatively it may be applied to the surface of the paper prior to the application of the CB coating as a separate step in some cases.

The invention will now be described by reference to examples which set out tests to compare and assess the archival characteristics of the resultant CFB grades made on a laboratory scale using a range of capsule coating compositions.

The additives used in the following examples are water based dispersions containing either alkyl ketene dimer (with or without dicyandiamide formaldehyde resin) stabilised by the addition of a cationic starch, or a modified rosin ester stabilised by the addition of a cationic starch.

The mechanism by which the enhancement is achieved when using these additives is not fully understood, but in the case of the rosin additive it is probable that a discontinuous covering of surface fibres of the substrate paper is formed with the molecules of rosin close enough to repel the solvent oil and thus preventing or significantly reducing migration of the solution into the paper.

In the case of the alkyl ketene dimer it is known that the central ketene ring reacts with the hydroxyl groups of the cellulose fibres to produce hydrophobic fatty acid chains which provide a resistance to migration. The formation of the linkage with the cellulose fibres is believed to be via beta-keto ester formations in accordance with the following mechanism:



wherein R = C₁₄ H₂₉ to C₁₈ H₃₇.

It is also known that alkyl ketene diene reacts with water to form waxy solids, and it is most likely that, during the application and drying of the modified capsule coating in accordance with the invention, a proportion of both of the above reactions occurs, thus producing a reduction in the permeability of the resultant dried coating film to migration of free oil and dyestuffs from prematurely broken capsules.

EXAMPLES

(a) PREPARATION OF SAMPLES

In each of the following examples the samples for comparative evaluation were prepared using as the substrate a continuous stationery paper of poor "through thickness porosity" previously coated on one side with a standard co-reactant CF coating.

All the samples were prepared using the above CF coated substrate and applying micro-capsules of a conventional colour-former solution to form a CB coating at the side of the above substrate paper opposite to that provided with the CF coating, using controlled conditions on a pilot coater.

In each Example a "Control" sample of the selected CF coated substrate paper was coated with a standard CB formulation for comparison with the "Test" samples, which were coated using a similar CB formulation to that of the "Control" but with the additives in accordance with the invention.

(b) ACCELERATED AGEING PROCEDURE (Humidity Cabinet)

The pilot coated "Control" and "Test" samples were initially sandwiched between two sheets of uncoated white card (150 gsm) and passed through a laboratory calender comprising two driven metal cylinders pre-set to a pressure of 25 bar. The result of this action is to rupture a high proportion of the capsules, resulting in the release of a quantity of free oil and colour-formers many times that experienced when handling or printing carbonless papers commercially.

The calendered samples were then suspended within a humidity cabinet pre-set to a controlled temperature of 55°C and a controlled Relative Humidity of 75° for a period of 48-72 hours. Upon removal of the samples from the cabinet the "L-value" (white to black scale) of the calendered area was determined using a fully calibrated colour measurement head and compared with the "L-value" of a corresponding un-aged control.

The combination of the higher than normal quantity of free oil and colour-formers released during the calendering operation together with the increased temperature and high humidity within the cabinet provides an accelerated ageing test equivalent to many years natural ageing.

Example 1

Pilot coater samples were prepared and evaluated using additions of a water based dispersion of a commercially available alkyl ketene dimer, a cationic

starch, and as the promoter, dicyandiamide formaldehyde in a dry ratio of AKD 12: cationic starch 3: promoter resin 6.

The alkyl radicals of the dimer were a mixture of predominantly C₁₄ to C₁₈ straight chain alkyl, and the dispersion contained 15.5 - 16.3 % solids, and had a density of 1.019 Kg/l at 20° C, a viscosity of 10 - 15 Cp at 25° C, and a pH in the range 2.5 - 4.0.

The additions used were as outlined below.

<u>Capsule Coating</u>	<u>Modified AKD/Starch</u>
100 parts dry (solid)	0 (control)
100 parts dry (solid)	1.5 parts dry/solid
100 parts dry (solid)	5 parts dry/solid

Humidity Cabinet Test Results - 66 hrs 55°C/ 75° RH

SAMPLE REF ADDITION	L-VALUE MEASUREMENT		L-VALUE DIFFERENCE
	UNAGED CONTROL	AGED SAMPLE	
CONTROL	90.60	81.94	8.66
1.5% ADDITION	88.34	84.08	4.26
5% ADDITION	90.40	87.99	2.41

The lower "L-values" of the aged samples are a direct indication of the background discolouration, the lower the "L-value" reading, the greater the background discolouration, and conversely the lower the "L-value" difference the less the background discolouration on ageing.

The above figures clearly show that the additives in the capsule formulation reduce the background discolouration.

Example 2

Example 1 was repeated using the same additive as used in Example 1 (i.e. alkyl ketene dimer/cationic starch combination modified by the addition of the promoter resin dicyandiamide formaldehyde) but in this example the range addition was extended as follows:

<u>Capsule Coating</u>	<u>Modified AKD/Starch</u>
100 parts dry (solid)	0 /control
100 parts dry (solid)	5 parts dry (solid)
100 parts dry (solid)	7.5 dry (solid)
100 parts dry (solid)	10.0 parts dry (solid)
100 parts dry (solid)	11.5 parts dry (solid)

In this example the cabinet ageing tests were evaluated after 48 hours.

Humidity Cabinet Test Results - 48 hours at 55°C/75° RH

SAMPLE REF ADDITION	L-VALUE MESUREMENT		L-VALUE DIFFERENCE
	UN-AGED CONTROL	AGED SAMPLE	
CONTROL	90.88	83.69	7.19
5% ADDITION	91.19	88.95	2.24
7.5 % ADDITION	91.45	90.03	1.42
10.0% ADDITION	91.03	90.23	0.80
11.5% ADDITION	90.85	90.04	0.81

It can be seen from the above results that the values obtained in Example 1 are repeated and that further enhancement is achieved by increased

additions up to 10% dry on dry. Additions greater than 10% dry on dry show no further improvement.

Example 3

The test procedure used for Example 2 was repeated using an alkyl ketene dimer/cationic starch dispersion without the promoter resin and with a dry alkyl ketene dimer / cationic starch ratio of 1 : 2, thus significantly reducing the proportion of alkyl ketene dimer in the dispersion.

In this case a different dimer was used, but again with a mixture of predominantly C₁₄ to C₁₈ straight chain alkyl chains, and the dispersion had a solids content of 30%, a viscosity of less than 400 mPas, and a pH in the range 3.7 - 4.7.

Additions used for this example were:-

<u>Capsule Coating</u>	<u>AKD/Starch</u>
100 parts dry (solid)	0 (control)
100 parts dry (solid)	5 parts dry (solid)
100 parts dry (solid)	7.5 parts dry (solid)
100 parts dry (solid)	11.5 parts dry (solid)
100 parts dry (solid)	13.5 parts dry (solid)

Humidity Cabinet Test Results - 48 hours/55°C/75° RH

SAMPLE REF ADDITION	L-VALUE MEASUREMENT		L-VALUE DIFFERENCE
	UNAGED CONTROL	AGED SAMPLE	
CONTROL	90.88	83.69	7.19
5% ADDITION	91.25	85.59	5.66
7.5% ADDITION	91.39	85.83	5.56
11.5% ADDITION	91.00	87.96	3.04
13.5% ADDITION	91.45	88.22	3.23

Here again the addition of the alkyl ketene dimer / starch blend shows an improvement compared to the Control sample. This improvement is less than that achieved using the alkyl ketene dimer dispersion used in Example 2, primarily due to the dilution of the proportion of alkyl ketene dimer added, but demonstrates that alkyl ketene dimer without promoter resin can produce an enhancement to substrates of poor "through thickness porosity".

Example 4

In this example an aqueous dispersion of a commercially available chemically modified rosin ester was used instead of an alkyl ketene dimer. The rosin was a penta erythritol hydrogenated rosin ester and the dispersion was stabilised by the addition of cationic starch, in the dry ratio range of 6 - 9 dry rosin ester : 1 dry starch, to give a total solids content of 30 - 40 %, with a viscosity of less than 150 mPas at 25° C, a density of 1.039 - 1.041 at 25° C and a pH in the range 7.3 - 8.3.

This dispersion was added to portions of standard capsule formula as follows:

<u>Capsule Coating</u>	<u>Rosin/Starch</u>
100 parts dry (solid)	0 (control)
100 parts dry (solid)	5 parts dry (solid)
100 parts dry (solid)	15 parts dry (solid)

Pilot coater samples were prepared and evaluated as outlined above.

Humidity Cabinet Test Results - 72 hours/55°C/75° RH

SAMPLE REF ADDITION	L-VALUE MEASUREMENT		L-VALUE DIFFERENCE
	UNAGED CONTROL	AGED SAMPLE	
CONTROL	91.18	79.12	12.06
5% ADDITION	91.40	83.70	7.70
15% ADDITION	91.53	87.08	4.45

The above results again show that the addition of a chemically modified rosin ester/starch dispersion reduces the background discoloration experienced when using a substrate of poor "through thickness porosity" to produce carbonless copy paper.

The above examples clearly establish that the inclusion of the coating additives in the capsule coatings used in the manufacture of carbonless copy paper significantly reduce the migration of the core material released from prematurely broken capsules through the paper substrate, thus reducing the tendency for the development of adverse effects during storage e.g. background discoloration, and therefore enables the satisfactory use of substrates of a lower

technical specification (i.e. exhibiting poor "through thickness porosity"). It is particularly to be noted that the invention makes it possible to utilise as a substrate for carbonless copy paper systems papers of grades previously considered quite unsuitable, including in particular xerographic photocopy grades, which are not required, by virtue of their intended use, to exhibit hydrophobicity.

While the alkyl ketene dimers used in examples 1 to 3 were based on saturated primary alkyl radicals with 14 to 18 carbon atoms, because of the commercial availability of such materials, it is nevertheless believed that, in general it would be possible to use such ketenes based on primary or secondary, saturated or unsaturated, alkyl chains of at least from 12 to 22 carbon atoms.

While the rosin used in example 4 was an ester, other suitable rosin derivatives are believed to include fortified rosins produced from the reaction of rosin acid with either fumaric acid or maleic anhydride, and which have generally similar properties as sizing agents.

Other sizing agents that it is believed could be employed include styrene-acrylic emulsion polymers.

While it is particularly preferred to incorporate such sizing agents in the CB coating composition, it would also be possible to treat the substrate paper with such sizing agents prior to the application of a conventional CB coating composition.

CLAIMS:

1. A coating composition for the preparation of CB coated carbonless copy paper characterised by the inclusion of an additive to increase resistance to penetration of the substrate paper by the non-aqueous solvents as used for the colour-formers employed in the CB coating.
2. A composition according to Claim 1 wherein the additive comprises a water based dispersion of a sizing agent which is such as to increase the hydrophobicity of a paper substrate when treated therewith.
3. A composition according to Claim 2 wherein the sizing agent is an alkyl ketene dimer.
4. A composition according to Claim 3 wherein the alkyl ketene dimer is based on alkyl radicals generally containing 12 to 22 carbon atoms.
5. A composition according to Claim 4 wherein the alkyl radicals containing 14 to 18 carbon atoms.
6. A composition according to any one of Claims 3 to 5 wherein the alkyl radicals are saturated primary alkyl.
7. A composition according to Claim 3 wherein the dimer is stabilised in emulsion form dispersion in a cationic starch.
8. A composition according to Claim 7 wherein the ratio of dry dimer to dry starch is between 10:1 and 1:2.

9. A composition according to Claim 3 wherein a cationic promoter is included to increase the rate of reaction between the dimer molecules and the anionic paper fibres of the substrate.

10. A composition according to Claim 9 wherein said promoter comprises dicyandiamide formaldehyde.

11. A composition according to Claim 10 wherein said promoter comprises a polyamide.

12. A composition according to Claim 10 wherein said promoter comprises a polyamine.

13. A composition according to Claim 9 wherein the dry ratio of said promoter to the dimer is in the range 1:10 to 1:1.

14. A composition according to Claim 2 wherein the sizing agent is a rosin derivative.

15. A composition according to Claim 14 wherein the rosin derivative is a rosin ester.

16. A composition according to Claim 15 wherein the rosin derivative is a penta erythritol hydrogenated rosin ester.

17. A composition according to Claim 14 wherein the rosin derivative is a fortified rosin produced by the reaction of rosin acid with either fumaric acid or maleic anhydride.

18. A composition according to Claim 14 wherein the dispersion includes a stabiliser.
19. A composition according to Claim 18 wherein the dispersion is stabilised with a cationic starch.
20. A composition according to Claim 19 wherein, the dispersion is stabilised with a wet strength resin.
21. A composition according to Claim 20 wherein said wet strength resin comprises a polyaminoamide.
22. A composition according to Claim 18, wherein said stabiliser is present in rosin to dry stabiliser ratio of 4:1 to 12:1.
23. A carbonless copy paper having a CB coating prepared from a composition as claimed in any one of the preceding claims.
24. A carbonless copy paper having a CB coating, characterised by the presence of an additive to increase the resistance to penetration of the paper by the non-aqueous solvents and used for the colour-formers employed in the CB coating.
25. A copy paper according to Claim 24 wherein the additive is a water based dispersion containing a sizing agent which is such as to increase the hydrophobicity of the paper substrate.

26. A copy paper according to Claim 25 wherein the sizing agent is included in a composition from which said CB coating is deposited.
27. A copy paper according to Claim 25 wherein the sizing agent is applied to the surface of the paper prior to the application of the CB coating.
28. A copy paper according to any one of Claims 25 to 27 wherein the sizing agent is an alkyl ketene dimer.
29. A copy paper according to any one of Claims 25 to 27 wherein the sizing agent is a rosin derivative.

INTERNATIONAL SEARCH REPORT

Int'l Application No
PCT/GB 99/02436

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B41M5/124 B41M5/132

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B41M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 576 176 A (WIGGINS TEAPE GROUP LTD) 29 December 1993 (1993-12-29) page 2, line 12 - page 3, line 15 examples ---	1-29
X	EP 0 491 487 A (WIGGINS TEAPE GROUP LTD) 24 June 1992 (1992-06-24) page 2, line 12 - line 36 page 3, line 12 - line 16 page 3, line 30 - line 40 examples ---	1-29
X	DE 43 12 854 A (FELDMUEHLE AG STORA) 27 October 1994 (1994-10-27) page 2, line 40 - line 47 page 2, line 66 - page 4, line 36 examples ---	1-25, 27-29
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

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